

A Collaborative Effort Between the U.S. EPA, the Food & Drug Administration, and the University of Arkansas to Develop a Method for the Determination of Parts-Per-Billion Levels of Carbamate Pesticide Residues in Eggs

Lynda Podhorniak, OPP/BEAD/ACB; Frank Schenck, James Hobbs, John Casanova, FDA/OR/ORA/SERL; and Dan Donoghue, University of Arkansas, Department of Poultry Science



Americans eat a lot of eggs. Each person in the U.S. eats about 250 eggs a year and there are an estimated 280 million egg laying hens in the U.S. producing about 80 billion table eggs a year.



Why is this of interest to the Environmental Protection Agency?

Because EPA scientists are determining dietary exposure estimates to pesticides for the American consumer and eggs are an important part of the American diet.



10% CARBARYL DUST

Carbamates are an important class of insecticides used in agriculture. Hens are directly treated with pesticides for external parasites. The carbamate carbaryl is routinely used for treating mites on hens by dusting or dipping.

So, chemists from the FDA and the EPA collaborated to develop an analytical method to detect very low levels of carbamate pesticides residues in eggs. The result of this collaboration is a new analytical method that can be used to quantify the amount of residual carbamate pesticides in eggs eaten by the American consumer down to levels as low as 2 parts per billion (ppb). The University of Arkansas dosed chickens with carbamate insecticides, then provided the eggs that were analyzed for residual pesticides.

SAMPLE PREPARATION



The sample extraction developed by the FDA:



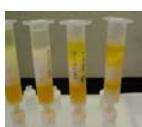
1. Eggs are extracted with 100 mL acetonitrile using a Polytron Homogenizer.

2. The acetonitrile extract is then centrifuged and dehydrated in a separatory funnel with Na_2SO_4 and MgSO_4 .

3. A portion of the dehydrated, filtered extract is transferred to a TurboVap® tube and evaporated to 1-2 mL.



The sample cleanup provided by the EPA:



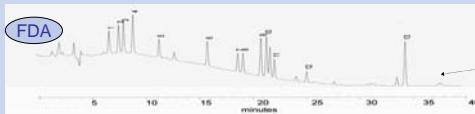
4. The concentrated extract is transferred to an aminopropyl SPE column which is topped with MgSO_4 & Na_2SO_4 . 1% Methanol/methylene chloride is eluted through the column and collected in a tube. 5. The extracts are then evaporated under a stream of N_2 .

6. 1.0 mL MeOH is added to the dried sample, which is filtered and transferred to an auto injector vial for instrumental analysis.

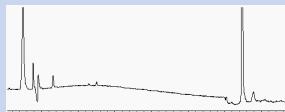
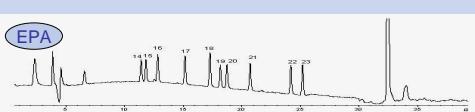
RESULTS

INSTRUMENTAL ANALYSIS

Both the FDA AND EPA EGG CHROMATOGRAMS show how feasible it is to see and quantify carbamates in eggs as low as 2 ppb using this carbamate method.



The egg control chromatogram below shows minimal interferences with the carbamate pesticides.



HPLC fluorescence chromatograms of FDA and EPA egg extracts fortified at 2.0 ppb. Peak identities are the following carbamates: (1) aldicarb sulfoxide, (2) aldicarb sulfone, (3) oxamyl, (4) methomyl, (5) 3-hydroxy carbofuran, (6) aldicarb, (7) propoxur, (8) carbofuran, (9) carbaryl, (10) 1-naphthol, (11) isopropcarb, (12) methiocarb, (13) piperonyl butoxide (14) thiophanox sulfoxide, (15) ethidamuron, (16) thiophanox sulfone, (17) butocarboxim, (18) metolcarb, (19) cloethacarb, (20) bendiocarb, (21) thiophanox, (22) fenobucarb, (23) promecarb.

Single-Comb White Leghorn hens were treated with carbaryl by scientists at the University of Arkansas and the eggs were analyzed by the FDA for incurred residues:

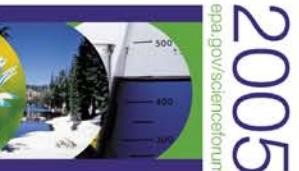


Dosing Method	Days after first dose	carbaryl ppb found	1-naphthol ppb found
Dusting	1	76	69
	2	54	33
	6	42	34
Spraying	2	14	5
	3	15	6
	4	23	6
Gelatin capsules	5	14	6
	1	186	243
	5	365	245
	6	401	232

Note that the levels of carbaryl and 1-naphthol (carbaryl metabolite) found in the eggs were well below the EPA legal limit or tolerance level of 500 ppb.



epascienceforum
Collaborative Science
for Environmental Solutions



2005
epascienceforum.org